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Comparison of gradient elution separations for organic and inorganic anions with chemically suppressed conductometric detection

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ABSTRACT

Gradient elution separations were developed for four pellicular and two macroporous resin based columns for the analysis of high-purity water. These elution techniques are applicable to both concentrator and loop injections and used sodium hydroxide and sodium tetraborate eluents with suppressed conductivity detection. The analytes of interest are fluoride, acetate, formate, pyruvate, chloride, nitrite, nitrate, bromide, sulfate and oxalate. These comparisons allow for the proper selection of column and eluent for part-per-billion (10^9) and sub-part-per-billion analysis of high purity water.

INTRODUCTION

As with all industry, managing escalating costs is of major concern. This is true as well for the power industry and especially true with nuclear power due to the size of the investment. With construction costs continuing to rise and increased regulatory requirements, power plants (fossil or nuclear fueled) now run into the billions (10^9) of U.S. dollars to construct. Because of these rising costs, emphasis is now being placed on increasing the life span of power plants through maintaining plant equipment integrity and material condition.

There are currently two major types of commercial nuclear reactors in the U.S.A.; boiling water reactors and pressurized water reactors. The boiling water reactor simply boils water in the reactor core to form steam to drive the turbine and electrical generator. As a result of this, non-volatile cationic and anionic impurities are concentrated in the reactor core and the recirculation piping causing stress corrosion and cracking. This is highly undesirable and must be minimized, due to the corrosive effect of these impurities.

The pressurized water reactor is different in that there are two separate systems or "loops" of water. The primary loop is pressurized to approximately 2250 p.s.i. which results in the heating of the reactor cooling water to 565°F without boiling. The secondary loop (low pressure) is where the steam is produced to drive the turbine and electrical generator. The interface between the primary and secondary loops is the

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steam generator. The secondary loop does not normally contain any radioactivity unless there is a material failure of the boundary. The steam generator functions solely as a heat exchanger through which the primary loop provides heat to the secondary loop to create steam and hence the term "steam generator".

Due to the function of the steam generator, an environment is created which promotes the concentration of impurities and the buildup of sludge, both of which have detrimental effects on system components and piping. These impurities initiate stress corrosion cracking and the buildup of sludge causing a loss of heat transfer efficiency and eventually causes a loss in generation capability.

If the sludge is not removed, the steam generators may degrade to the point where they need to be replaced at a cost of hundreds of millions of U.S. dollars, not to mention the costs associated with the loss of revenue due to the downtime required to replace the steam generators. This can be prevented by maintaining impurity levels as low as possible and by taking an active role in identifying the sources of impurities. Ion chromatography has become increasingly important by allowing for the detection and measurement of extremely low levels of impurities and determination of the type of impurity in the high purity water used by the power industry.

It was rapidly recognized in the initial developing stages of high-performance liquid chromatography (HPLC), that like gas chromatography, it was a powerful technique for the separation and determination of organic analytes. During this period few chromatographic studies were devoted to inorganic separations, and other methods (electrode, spectroscopic) remained the major choice when solving inorganic analytical problems. This changed in 1975 when Small *et al.* [1] published their classic paper on high-performance ion-exchange separation with post column suppression, generally referred to as modern day ion chromatography (IC). This technique allowed the separation and determination of common inorganic anions and cations at ppm levels under isocratic conditions.

Additional developments in IC have continued into other applications where IC plays a role, concurrently increasing sensitivity of analysis through the use of "stripping" (concentrator) columns. As this separation technology progressed through the use of new eluents and more efficient columns, it was recognized that other species were eluting and co-eluting with the primary species of interest. To determine what these species were another instrument had to be set up under different conditions to resolve the problem. An example of this in the power industry is the presence of organic anions (most notably acetate and formate) which caused elution problems with fluoride and chloride in the standard carbonate–bicarbonate eluent conditions often used with chemically suppressed conductometric detection.

A means of resolving these coelution problems presented itself in 1986 when the Dionex Corporation introduced a new pump to fill the perceived need for gradient IC. Gradient elution begins with an eluent of low ionic displacement capabilities and progresses either gradually or in a series of steps to an eluent of greater ionic displacement capabilities to cope with species of widely varying affinities (capacity factors, k') for the stationary phase. In these cases, eluent conditions that favor the resolution of the most weakly retained species are often unsuitable for the more strongly held ions in that they lead to frustratingly long elution times and poor efficiencies. The reverse is also true in that using a stronger eluent, while it may elute the strongly held ions in a reasonable time, typically compromises the resolution of early eluting ions. In these cases gradient elution is the means for solving the problem.

Suppressed conductometric methods, since they reduce the background conductance of the eluent, are readily adapted to the use of gradient eluent methodology. With the advent of new high-capacity membrane suppression devices, gradient elution can be achieved in anion analyses. In this regard, sodium hydroxide as an eluent is becoming increasingly important in gradient analysis. From the suppression standpoint sodium hydroxide is the ideal eluent since its product is water, but its low ion-exchange affinity has limited its usefulness in the past [2].

While gradient elution is an effective way of handling ions of diverse affinities, it is not without its problems. The most noteworthy of these is the buildup of eluent impurities in the separator column and later release as the eluent concentration is increased. This problem has been examined by Rocklin *et al.* and measures have been suggested to alleviate it [3], such as the use of trap columns. It is therefore apparent that eluent preparation technique plays a vital role in gradient elution methodology. However, the purer the reagent the less this becomes a problem.

There has been some work in gradient IC that is useful for the power industry [4,5]. However, most of this work is based on a continuous gradient profile. Due to the continuously changing baseline, problems may be encountered with the reproducibility and resolution in the low ppb range that is normally required by the power industry. The work presented in this study focuses on the use of a single step gradient profile to minimize baseline changes thereby increasing reliability and reproducibility. This is also done so that laboratories without gradient eluent pumps may also benefit by the usefulness of gradient analysis and apply it without having to purchase additional equipment.

This study will concentrate on the determination of organic and inorganic anions in a single chromatographic run, utilizing pellicular and macroporous resin based columns in conjunction with hydroxide and tetraborate eluents, using suppressed conductivity detection. The methods presented here are applicable to loop injections in the ppm range also.

EXPERIMENTAL

Instrumentation

All chromatography in this study was performed using a Dionex (Sunnyvale, CA, U.S.A.) series 2020i ion chromatograph, except that a gradient pump was substituted for the analytical pump. The dual-channel ion chromatograph was interfaced with a multichannel data-and-control system consisting of a Autoion 450 Dionex computer interface, Dell (Austin, TX, U.S.A.) System 200 desk-top computer with a 40-megabyte hard drive, a 5.25-in. floppy drive, and a Epson FX850 printer (Seiko Epson, Japan). Sample delivery was accomplished with a Dionex ASM autosampler in all instances but two. In these two cases, a Dionex DQP pump was used due to the normal higher backpressure expected when using the AS5A and Omnipac PAX 500 column sets. Dionex AI450 software provided data acquisition, data reduction and control of the ion chromatograph. A diagram of the system is shown in Fig. 1.

Reagents and standard solutions

Gradient IC is still in its infancy. Consequently, eluent purity, analogous to

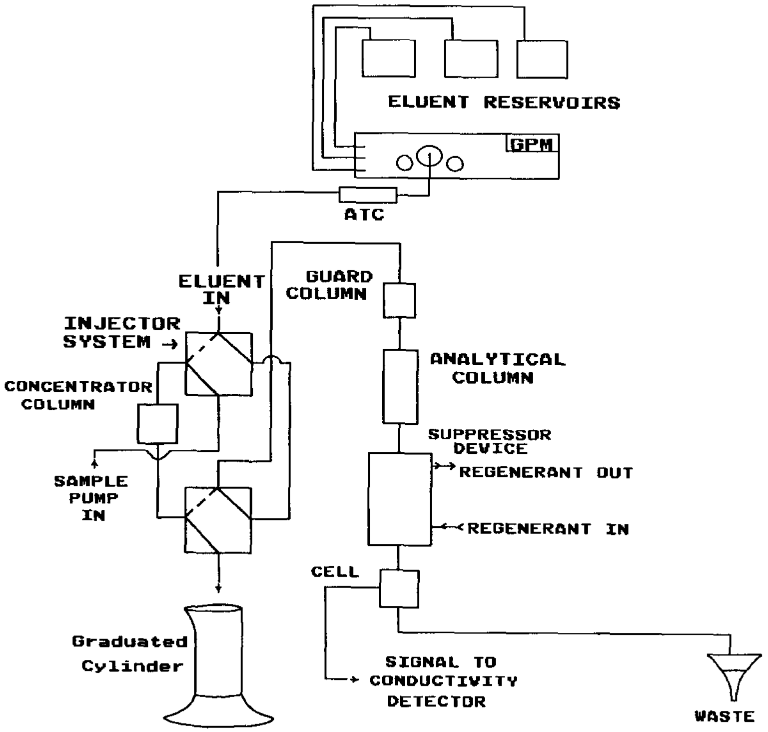

Fig. 1. Schematic of chromatographic system. ATC = Anion trap column.

HPLC-grade solvents, is an issue of vital importance. Reagent-grade chemicals were used throughout this study with the exception of the sodium hydroxide used for an eluent. Suprapure sodium hydroxide available from VWR (Houston, TX, U.S.A.) was used in hydroxide eluent preparation due to the minimal contaminant content. The water used was plant-prepared demineralized water passed through a Barnsted Nanopure (Barnsted/Thermolyne, Dubuque, IA, U.S.A.) water system to ensure 18-MΩ quality. Due to the rapid deterioration of the organic anion standards below 10 ppm, they were prepared from stock standards as needed. The stock 1000-ppm standards were kept refrigerated when not in use. The sodium hydroxide eluent concentrate was prepared by dissolving 4.00 grams in 600 ml of high-purity water, diluted to 1000 ml to yield a 0.1 *M* solution and degassed to minimize carbonate and sealed under a nitrogen overpressure blanket to prevent carbonate uptake. The sodium tetraborate eluent was prepared in the same fashion with 38.1 g of sodium borate decahydrate to yield a 0.1 *M* solution. Heat had to be applied to affect dissolution of the tetraborate.

Table I lists the standards and amount of reagent needed to make a 1 l of 1000-ppm stock standard [6] of each anion. The salts were dried at 105°C for 4 h prior to use with the exception of the organic anions which would break down under these conditions. Standard solutions were stored in dedicated and precleaned Pyrex lab-

TABLE I

1000 ppm STANDARD SOLUTIONS

Standard	Reagent	Amount required (g)
Fluoride	Sodium fluoride	2.2100
Acetate	Sodium acetate	1.6585
Formate	Sodium formate	1.5111
Pyruvate	Sodium pyruvate	1.2614
Chloride	Sodium chloride	1.6484
Nitrite	Sodium nitrite	1.4998
Bromide	Sodium bromide	1.2877
Nitrate	Sodium nitrate	1.3707
Sulfate	Sodium sulfate	1.4791
Oxalate	Sodium oxalate	1.5227

ware to minimize anion leaching. The organic standards were refrigerated when not in use to prevent deterioration.

Columns

To reduce eluent impurities, an anion trap column (ATC as shown in Fig. 1) was placed on the outlet of the eluent pump going to the chromatographic module and before the injection valve. The trap column function is to "smear out" eluent impurities by preventing the impurities from building up on the analytical column and eluting as a peak as the gradient program is run. The analytical column sets used in this study were all manufactured by Dionex and are presented in Table II. Table III lists the general column characteristics of the columns used in this study.

Procedures

In the secondary loop of power plants, cation additives such as ammonia, morpholine and hydrazine are added to control pH and oxygen. These cationic additives are removed by passing a sample through a cation-exchange column to remove them prior to the analysis of anions. This effectively returns the samples to a "high-purity" condition. The results presented below focus on the analysis of anions in

TABLE II

COLUMN SETS

Concentrator	Guard	Analytical
AG4A	AG4A	AS4A
AG5	AG5	AS5
AG5A	AG5A	AS5A
AG9	AG9	AS9
PAX 100 Guard	PAX 100 Guard	PAX 100 Analytical
PAX 500 Guard	PAX 500 Guard	PAX 500 Analytical

TABLE III

COLUMN CHARACTERISTICS

Analytical column·	Substrate (μM)	Latex X-link (%)	Capacity (μequiv./column)	Hydrophobic nature
AS4A	15	0.5	20	Medium–low
AS5	15	1.0	20	Low
AS5A	5	4.0	35	Low
AS9	15	[a]	35	Medium–low
PAX 100[b]	8.5	4.0	40	Hydrophilic
PAX 500[c]	8.5	4.0	40	Hydrophilic

[a] Contains a multiple functionality cross-linking which is different than other columns. This column is acrylic based.

[b] Latex diameter 60 nm, surface area <1 m^2/g.

[c] Latex diameter 60 nm, surface area 300 m^2/g.

high-purity water without the additives. All anions were detected with a full scale of 30 μS output range. Volumes of 10 ml of the mixed standard were concentrated in all cases. Standards were analyzed singly under the conditions listed in Table IV to insure optimum resolution and to determine the corresponding retention times. The standards were then run all together by performing three sequential injections at the 5-, 25- and 50-ppb levels and averaging the values at each level for the calibration. All calibration data revealed linearity (r^2) values of at least 0.99. The precision of the analytical methods presented were determined by performing 25 replicate analysis at the 25-ppb level containing the ten anions of interest. These replicate analyses were used for the determination of relative standard deviations (R.S.D.) and ensured accurate reproducibility.

RESULTS AND DISCUSSION

The separation and quantification of organic and inorganic anions was

TABLE IV

GENERAL CONDITIONS

Concentrator column	Analytical column	Eluent type	Eluent flow (ml/min)	Gradient profile
AG4A	AS4A	NaOH	2.0	3.5 m*M*/35 m*M*
AG4A	AS4A	Tetraborate	2.0	5.0 m*M*/30 m*M*
AG5	AS5	NaOH	1.5	2.0 m*M*/25 m*M*
AG5	AS5	Tetraborate	1.5	3.0 m*M*/25 m*M*
AG5A	AS5A	NaOH	1.5	3.0 m*M*/60 m*M*
AG9	AS9	Tetraborate	2.0	1.0 m*M*/30 m*M*
PAX 100	PAX 100	NaOH–methanol	1.0	5 m*M* NaOH–5% methanol
Guard	Analytical	NaOH–methanol	1.0	80 m*M* NaOH–10% methanol
PAX 500	PAX 500	NaOH–methanol	1.0	1 m*M* NaOH–2.5% methanol
Guard	Analytical	NaOH–methanol	1.0	60 m*M* NaOH–2.5% methanol

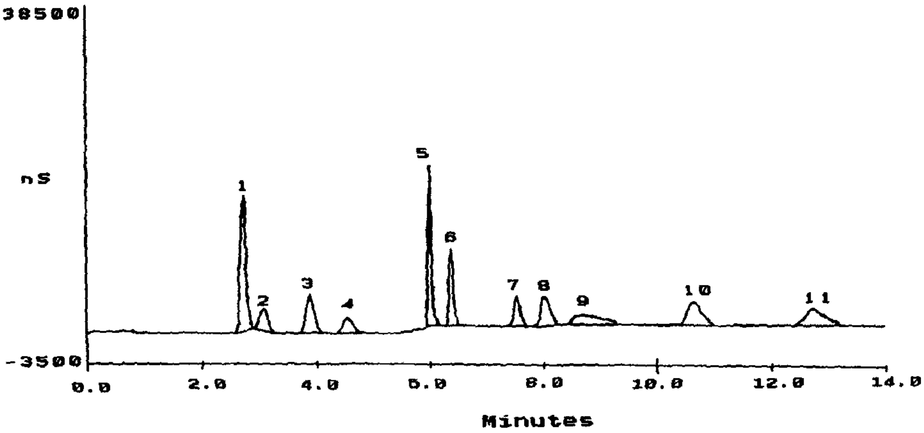

Fig. 2. AS4A separation using sodium hydroxide. Peaks: 1 = fluoride; 2 = acetate; 3 = formate; 4 = pyruvate; 5 = chloride; 6 = nitrite; 7 = bromide; 8 = nitrate; 9 = carbonate; 10 = sulfate; 11 = oxalate. Gradient program: 3.5 m*M* NaOH stepped to 35 m*M* NaOH 4 min after injection. Regenerant: 20 m*M* sulfuric acid at 5.0 ml/min.

achieved successfully on all the analytical columns with the exception of the AS9 column. The acetate peak coeluted with the fluoride peak and the formate and pyruvate peaks coeluted. This may be attributed to the fact that the AS9 column is the only acrylic based column tested and was designed to perform U.S. Environmental Protection Agency drinking water analyses. Example chromatograms are presented in Figs. 2–9 for each method developed.

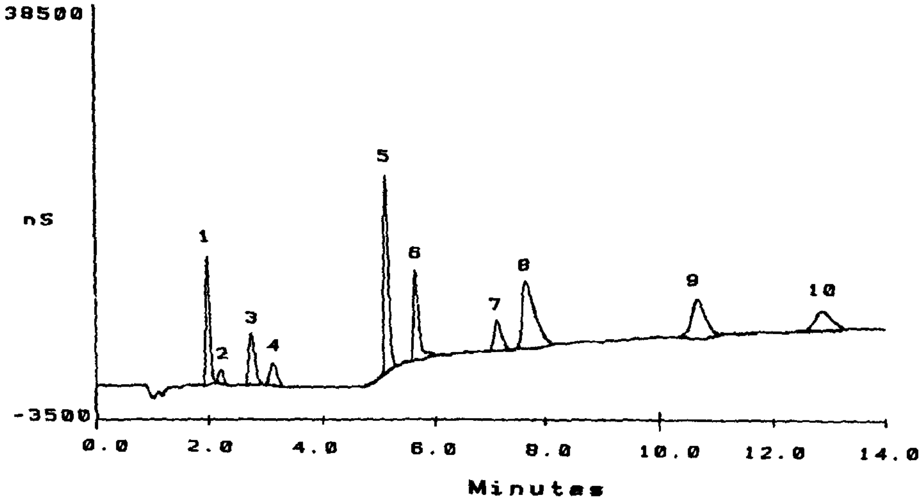

Fig. 3. AS4A separation using sodium tetraborate. Peaks: 1 = fluoride; 2 = acetate; 3 = formate; 4 = pyruvate; 5 = chloride; 6 = nitrite; 7 = bromide; 8 = nitrate; 9 = sulfate; 10 = oxalate. Gradient program: 5.0 m*M* tetraborate stepped to 30 m*M* tetraborate 4 min after injection. Regenerant: 20 m*M* sulfuric acid at 10.0 ml/min.

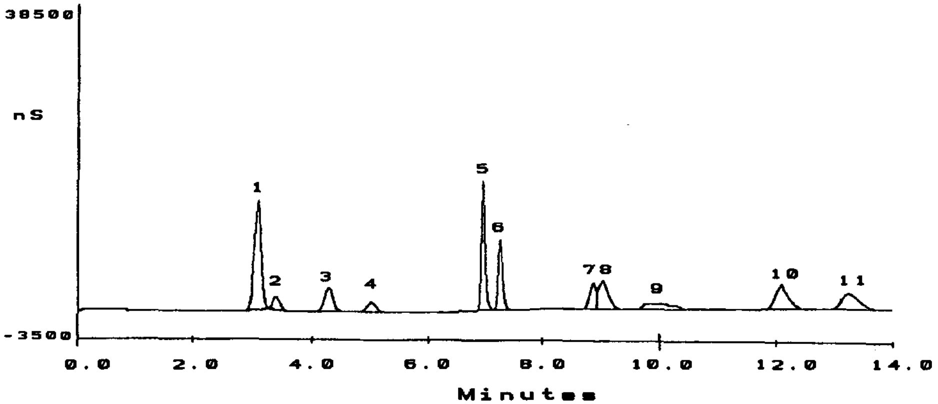

Fig. 4. AS5 separation using sodium hydroxide. Peaks as in Fig. 2. Gradient program: 2.0 m*M* NaOH stepped to 25 m*M* NaOH 4 min after injection. Regenerant: 20 m*M* sulfuric acid at 5.0 ml/min.

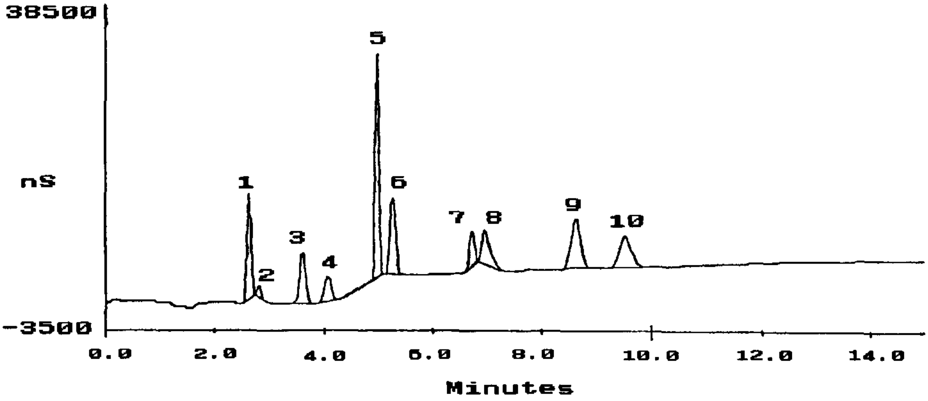

Fig. 5. AS5 separation using sodium tetraborate. Peaks as in Fig. 3. Gradient program: 3.0 m*M* tetraborate stepped to 25 m*M* tetraborate 3 min after injection. Regenerant: 20 m*M* sulfuric acid at 10.0 ml/min.

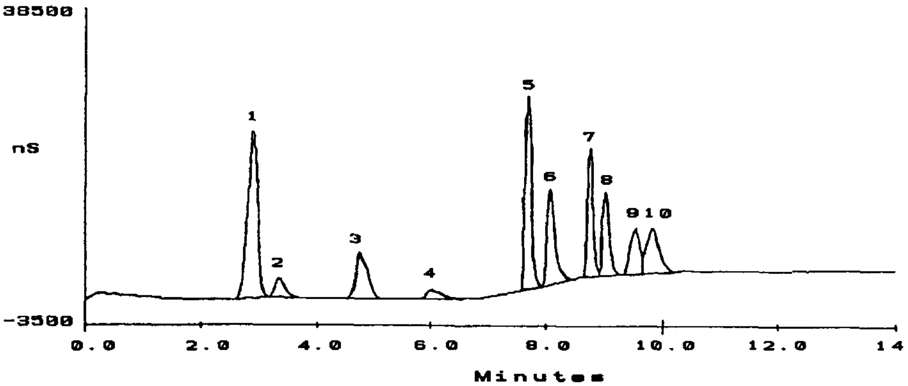

Fig. 6. AS5A separation using sodium hydroxide. Peaks: 1 = fluoride; 2 = acetate; 3 = formate; 4 = pyruvate; 5 = chloride; 6 = nitrite; 7 = sulfate; 8 = oxalate; 9 = bromide; 10 = nitrate. Gradient program: 3.0 m*M* NaOH stepped to 60 m*M* NaOH 5 min after injection. Regenerant: 20 m*M* sulfuric acid at 5.0 ml/min.

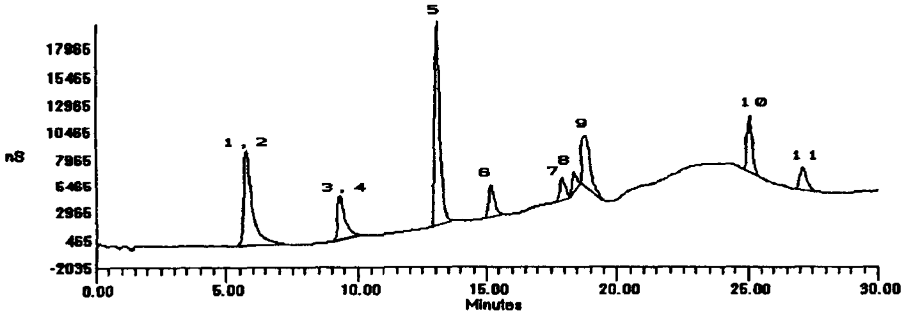

Fig. 7. AS9 separation using sodium tetraborate. Peaks: 1 = fluoride; 2 = acetate; 3 = formate; 4 = pyruvate; 5 = chloride; 6 = nitrite; 7 = bromide; 8 = unknown; 9 = nitrate; 10 = sulfate; 11 = oxalate. Gradient program: 1.0 m*M* tetraborate ramping to 30 m*M* tetraborate starting 5 min after injection and finishing 25 min after injection. Regenerant: 20 m*M* sulfuric acid at 10.0 ml/min.

To assist in column and method evaluation the capacity factors of each organic and inorganic anion were calculated using the equation [7]: $k' = (t_R - t_0)/t_0$, where t_R is the retention time and t_0 the retention of an unretained compound and are presented in Table V.

To further assist in column evaluation the theoretical plates (efficiency) were calculated for each anion using the equation [7]: $N = 16(t_R/w)^2$, where w is the peak width. The values obtained are presented in Table VI.

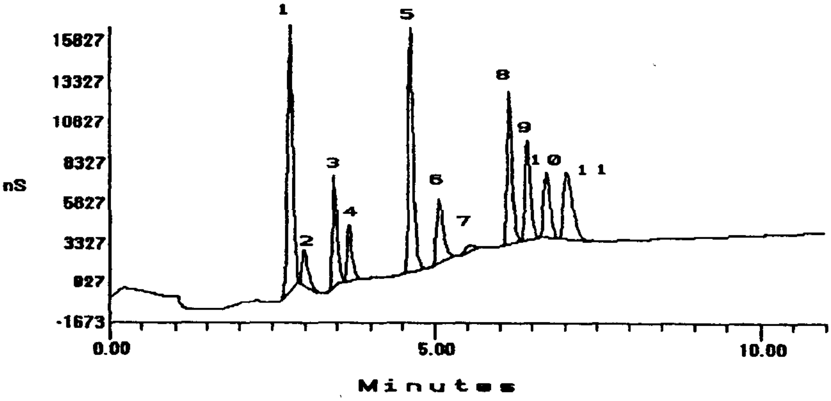

Fig. 8. Omnipac PAX 100 separation using sodium hydroxide. Peaks: 1 = fluoride; 2 = acetate; 3 = formate; 4 = pyruvate; 5 = chloride; 6 = nitrite; 7 = carbonate; 8 = sulfate; 9 = bromide; 10 = nitrate; 11 = oxalate. Gradient program: eluent 1: 100 m*M* NaOH; eluent 2: methanol–water (50:50); eluent 3: deionized water. Regenerant: 20 m*M* sulfuric acid at 5.0 ml/min.

Time (min)	Eluent 1 (%)	Eluent 2 (%)	Eluent 3 (%)	Comments
0.0	5.0	10.0	85.0	Equilibrate and load
4.0	5.0	10.0	85.0	Start gradient ramp
6.0	40.0	10.0	50.0	Inject sample
6.1	80.0	20.0	0.0	Step gradient
17.0	80.0	20.0	0.0	End run

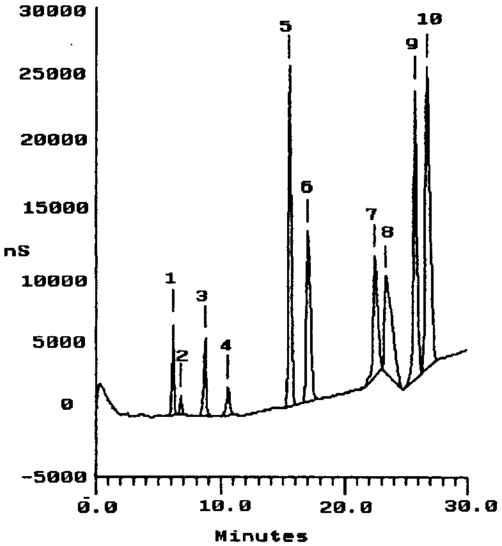

Fig. 9. Omnipac PAX 500 separation using sodium hydroxide. Peaks: 1 = fluoride; 2 = acetate; 3 = formate; 4 = pyruvate; 5 = chloride; 6 = nitrite; 7 = bromide; 8 = nitrate; 9 = sulfate; 10 = oxalate. Gradient program: eluent 1: 2 m*M* NaOH; eluent 2: methanol–water (50:50); eluent 3: 200 m*M* NaOH. Regenerant: 40 m*M* sulfuric acid at 5 ml/min.

Time	Eluent 1 (%)	Eluent 2 (%)	Eluent 3 (%)	Comments
0.0	95.0	5.0	0.0	Equilibrate and load
5.0	95.0	5.0	0.0	Inject
10.0	95.0	5.0	0.0	Start gradient ramp 1
22.0	80.0	5.0	15.0	Start gradient ramp 2
35.0	55.0	5.0	40.0	End run

As can be seen from Tables V and VI the most effective column for use in the power industry is the PAX 100 with the NaOH and methanol eluent due to its efficiency and the rapidity of the separations. Concerning the use of the Omnipac PAX 100, it was found to function better if it was only re-equilibrated for 4 min while the next sample was loading.

Using Figs. 2, 4, 6, 8 and 9 as guidelines, it can be seen that there is very little baseline disturbance. This fact is due to the use of NaOH as an eluent with the use of an anion trap column (ATC). During this study, a single ATC was used for the NaOH separations without having to be regenerated, and the baseline conductivities ranged from 1–3 μS. A new ATC was used for the tetraborate separations and had to be regenerated every 40–50 samples, with the baseline conductivities running 6–10 μS.

One other point that should be noted is the use of an autosampler. A Dionex ASM was used in all cases with the exception of the AS5A and PAX 500 column sets. The backpressure from these columns would not allow for the use of the autosampler. A new autosampler needs to be designed to allow for higher backpressures. The Dionex ASM autosampler is satisfactory if only 5 ml are being concentrated, but is unacceptably slow (1 ml/min). This makes for long analysis times, where it takes almost as long to concentrate 10 ml of sample as it takes for the analysis.

TABLE V

CAPACITY FACTORS (k')

Column/eluent	Fluoride	Acetate	Formate	Pyruvate	Chloride	Nitrite	Bromide	Nitrate	Sulfate	Oxalate
AS4A/NaOH	2.47	2.89	3.95	4.79	6.68	7.16	8.68	9.26	12.58	15.32
AS4A/tetraborate	0.81	1.52	2.14	2.57	4.95	5.57	9.24	9.90	11.33	13.90
AS5/NaOH	2.17	2.52	3.43	4.18	6.13	6.42	8.04	8.33	11.46	12.67
AS5/tetraborate	1.22	1.33	2.04	2.41	3.15	3.41	4.26	4.85	5.48	6.96
AS5A/NaOH	2.40	2.95	4.50	5.15	8.20	8.65	10.4	10.75	9.35	9.65
AS9/tetraborate	3.06	3.06	5.56	5.56	8.19	10.19	11.44	12.13	16.44	17.88
PAX 100/NaOH–methanol	1.52	1.75	2.13	2.35	3.23	3.62	4.85	5.12	4.60	5.42
PAX 500/NaOH–methanol	0.71	0.97	2.43	4.64	5.17	5.83	8.03	8.46	9.33	9.79

TABLE VI

THEORETICAL PLATES

Column/eluent	Fluoride	Acetate	Formate	Pyruvate	Chloride	Nitrite	Bromide	Nitrate	Sulfate	Oxalate	Mean
AS4A/NaOH	4600	1450	2300	2000	21 300	24 300	15 000	9600	10 800	6000	9750
AS4A/tetraborate	2300	2900	1900	2600	15 900	8500	7600	3900	5400	8100	5900
AS5/NaOH	2600	7100	4800	3700	28 200	31 000	11 400	11 900	13 600	11 400	12 500
AS5/tetraborate	3600	4100	3000	3800	13 000	6400	9000	6400	9600	7500	6650
AS5A/NaOH	1200	1600	3000	2900	15 400	9600	8500	8900	20 000	21 100	9200
AS9/tetraborate	N/A[a]	N/A	N/A	N/A	22 600	22 900	N/A	N/A	91 900	56 400	N/A
PAX 100/NaOH–methanol	6100	5300	8100	11 600	14 600	9800	28 200	17 300	32 600	11 100	14 500
PAX 500/NaOH–methanol	11 300	9700	9500	10 700	20 600	12 300	19 100	6800	47 600	21 900	16 950

[a] N/A = Not applicable.

TABLE VII

RELATIVE STANDARD DEVIATION AT 25 ppb

Column/eluent	R.S.D. (%)									
	Fluoride	Acetate	Formate	Pyruvate	Chloride	Nitrite	Bromide	Nitrate	Sulfate	Oxalate
AS4A/NaOH	3.2	3.9	2.2	4.8	1.9	2.1	3.3	3.4	4.6	5.4
AS4A/tetraborate	4.4	5.6	2.4	5.3	2.4	2.9	4.7	4.1	5.3	6.3
AS5/NaOH	2.2	3.1	2.4	5.1	1.8	2.5	4.8	5.1	4.3	4.9
AS5/tetraborate	4.1	4.3	2.3	3.9	1.9	2.8	4.3	5.1	4.6	5.4
AS5A/NaOH	3.1	3.8	2.9	6.4	3.4	3.6	6.6	6.9	5.3	5.8
AS9/tetraborate	N/A	N/A	N/A	N/A	4.6	3.8	7.9	8.4	4.2	4.6
PAX 100/NaOH–methanol	4.8	4.7	2.3	2.4	1.6	1.8	2.6	2.8	2.4	3.2
PAX 500/NaOH–methanol	3.4	4.1	2.4	2.5	1.8	2.1	3.8	4.1	2.5	3.1

The R.S.D. values for the methods developed are presented in Table VII. As listed in the table, better R.S.D. values were found for the NaOH eluents than with the tetraborate eluents. This is due to less baseline disturbance and lower eluent conductivities. This also plays a role in higher sensitivities. The less baseline disturbance and the lower the background conductivity, the higher the sensitivity will be for ions of interest. However, tetraborate eluent has been shown to be beneficial in reducing interferences for analysis of anions in matrices containing boric acid.

CONCLUSIONS

The need for gradient IC is obvious from the diversity of demands made on the power plant chromatographer. In particular, the ability to quickly screen a sample for organic and inorganic anions that cause performance losses can potentially save millions of dollars. Screening for a diverse range of ions in a single ion chromatographic run is a lofty goal. The methods presented here are also applicable to loop injections for the study of anions in the ppm range.

It is recommended that for analysis of organic and inorganic anions that the PAX 100 columns be used with the conditions listed with Fig. 9. It has also been seen that with the higher efficiency of the PAX 100 that lower limits of detection for on-line ion chromatographs may be achieved at the 5 part-per-trillion (10^{12}) level by concentrating only 30 ml of sample. For those chromatographers only interested in the separation of the common inorganic anions (F^-, Cl^-, SO_4^{2-}), the AS5 column with an 25 m*M* NaOH eluent will yield excellent results in only a four minute run. As a direct result of this study, the PAX 100 method developed has been adapted to the on-line ion chromatographs employed in monitoring the secondary loop at the South Texas Project.

The Omnipac PAX 500 column appears to have a lot of potential due to its ability to perform both anion-exchange and reversed-phase separations in a single injection. Future work with this column should prove very interesting. Other work also needs to be focused on an advanced design autosampler for use with large volumes (10–25 ml) of sample so that they may be concentrated at a faster rate (3–4 ml/min).

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